STUDY ON CONTROL OF MESOPHASE SPHERULITS DIAMETERS

Hu Zijun Ling Licheng Liu Lang Zhang Bijiang
Institute of Coal Chemistry, Chinese Academy of Science,
P.O.B. 165, Taiyuan, Shanxi, 030001,China

Introduction

Meso-Carbon Microbeads (MCMBs) have been studied extensively as functional materials such as super absorptive material, selfsintering material, column packing material of HPLC, electrode material of Li^+ cell, catalyst carrier etc.[1,2].

Although many preparation methods of MCMB have been developed[3,4], there still remained some problems such as size distribution of MCMB to be improved.

Naphthalene pitch was used as raw material to prepared MCMB. In order to get MCMB with narrow size distribution, parts of toluene soluble fraction of the pitch were removed by extraction before pyrolysis and additive of ferrocene was also tested.

Experimental

Naphthalene pitch (Pitch A) was obtained by the heat treatment of naphthalene oligomers, which was prepared from pure naphthalene using ZrO_2/SO_4^{2-} as catalyst[5], at 450℃for 3hr under N_2 flow. Pitch B was prepared by removing 9.5wt% of toluene soluble fraction from Pitch A. Pitch A' and B' were obtained by adding 2wt% of ferrocene into Pitch A and B respectively. Some properties of these pitches are summarized in Table 1.

All of the pitches were heated treated at 380℃ with a heating rate of 20 ℃ /min under a hot-stage polarized microscope. The holding time at 380℃ was 10min for Pitch A and B, while that was 2min for Pitch A' and B'.

Results and Discussion

The polarized microscopies of MCMB derived from pitches are shown in Fig. 1-4. Compared with that of Pitch B, the MCMB in Pitch A shows smaller size. This might be due to the Pitch A has more BS fraction (44.1wt%), which are small molecules preventing the coalescence among mesophase spheres. Both of the MCMB in Pitch A and B have a little wide size distribution(Fig. 1 and 2)

Adding ferrocene into pitches not only prevented the coalescence of mesophase spheres efficiently, but also increased the yields of MCMB significantly in comparison with pitches without ferrocene. It seemed the ferrocene acts as active site during the formation of MCMB. Removal of some toluene soluble fraction from pitch can further increased the yield of MCMB in the presence of ferrocene(Fig. 4). Moreover MCMB with very unique diameter has been obtained by the adding of 2wt% ferrocene into pitches. The present work revealed that the diameter and size distribution as well as yield of MCMB could be controlled by adjusting the content of pitch fraction and adding

additive of ferrocene except for the pyrolysis conditions, such as terminal temperature and holding time, etc..

References

1. Kasuht et al., 19th Biennial conference on Carbon, 88(1989)
2. J. yamaur et al.,Extended Abstracts of 6th International Meeting on Lithum Batteries,1992.
3. M. Kodama et al., Carbon 28, 199(1990)
4. S. H. Yoon et al., Carbon 30, 781(1992)
5. Z. J. Hu et al., 22nd Biennial Conference on Carbon, San Diego, 258, 1995.

Table1. Some essential properties of pitches

No.	TS wt%	TI-PS wt%	PI wt%	S.P. ℃	A.C. Vol%	A.T.
pitch A	44.1	23.7	32.2	202	20	Scatter distribution of spherulits
pitch B	36.2	27.8	36.0	238	55	Scatter distribution of spherulits
pitch A'	44.1	23.7	32.2	202	20	spherulits with unique size ca. 5μm
pitch B'	36.2	27.8	36.0	238	55	spherulits with unique size ca. 11μm

★ TS:toluene soluble fractions, TI-PS:toluene insoluble but pyridine soluble factions, PI:pyridine insoluble fractions, S.P.:softening point, A.C.:anisotropic content, A.T.:anisotropic texture.

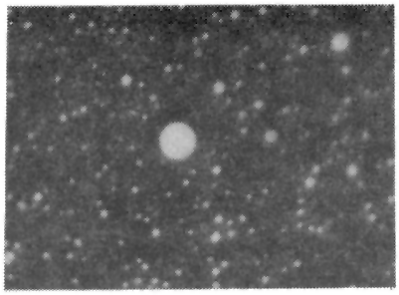

Fig.1 pitch A

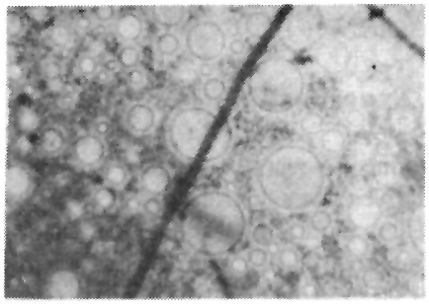

Fig.2 pitch B

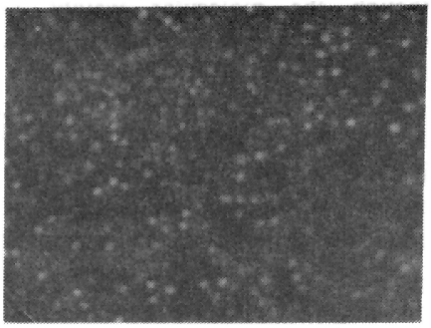

Fig.3 pitch A'

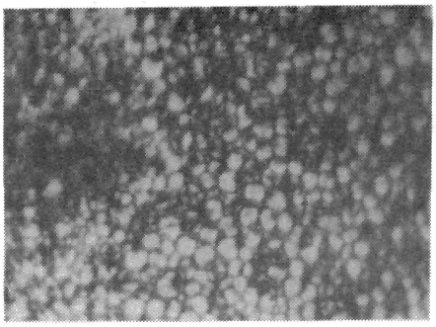

Fig.4 pitch B'